Synchrotron Radiation Instrumentation
Collaborative Access Team

SRI CAT NEWSLETTER

Vol. 3, No. 2 **January 1997**

From the desk of the Program Director:

It is a well established tradition at the end of the year to summarize the good things that happened and try to forecast events for the forthcoming year. Nineteen ninety six was extremely critical for SRI CAT because of the beginning of the transition from the construction phase to operation. Most of the construction activities were successfully accomplished this year. Sector 1 construction is nearly 100% completed, at Sector 2, the level of completion is close to 70-75%, and Sector 3 is about 85-90% done. Due to the solid and dedicated performance of each SRI CAT member, as well as the strong support of the APS staff and outside contractors, the planned scope of the construction of the three sectors will be finished in the next few months.

In the meantime, SRI CAT technical staff has used every opportunity during the experimental runs of the last year to test new equipment developed under its strategic instrumentation plans, and to conduct state-of-the-art experiments at the APS. Several highlights below reflect only some selected achievements in the instrumentation development areas.

In one of the most critical areas for the whole APS community, high heat load optics, several important results have been achieved. Both thin and thick cryogenically cooled silicon crystals have shown quite small thermal broadening at closed undulator gaps. Also, a diamond double crystal monochromator showed no thermal-induced broadening of the 2-3 arc-second-wide rocking curve. (This width is due to imperfect nature of the diamonds.) In addition, the first set of successful tests have been performed with a side-cooled high heat load mirror and water-cooled U-shape monochromator.

Developments in hard x-ray instrumentation were significant because of the remarkable performance of Undulator A in the 80-100 keV energy range, and successful optics tests for use in 0.1 a.u-resolution Compton scattering . Also, a dual-helicity phase retarder that was designed to produce circularly polarized x-rays at 86 keV has shown very good performance that was predicted theoretically.

A novel monochromator with 0.8-meV resolution at 14.4 keV energy has been developed, successfully tested, and used for several high resolution inelastic nuclear resonant x-ray scattering experiments.

The microfocusing technique has been advanced to better than 0.5 micron spatial resolution and record efficiency for 8 keV x-rays.

Overall, achievements in development of instrumentation by SRI CAT are quite significant and, in many cases, represent state-of-the-art techniques.

As a very young and healthy organization, SRI CAT has grown rapidly in 1996, and all of us were very happy to accept into our family new members from faraway Australia and close-by Purdue & Assoc. But, a growing family typically brings a strong demand for a real estate. Our case is no exception. Therefore SRI CAT and XFD management are considering the expansion beyond the three existing sectors. In January, new plans for the development of a fourth sector will be presented before the APS Proposal Evaluation Board. It is necessary to point out that this expansion request is not only due to the increase in the SRI CAT membership, but also because of the quality of the x-ray polarization program that has developed rapidly in the last several years. This program would require two insertion devices: Undulator A for hard x-rays and a new special helical undulator for the intermediate energy range. With new crystal optics instrumentation and special insertion devices, the SRI CAT polarization program would be the most advanced and comprehensive program of this sort at synchrotron radiation facilities around the world.

In January 1997, the next SRI CAT meeting will take place. This interesting meeting program will include reports on our accomplishments as well as discussions of future plans. I would like to take this opportunity to welcome all participants of the forthcoming meeting and to wish all of them a happy and successful New Year

E. Gluskin, SRI CAT Director

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Recent Nuclear Resonant Scattering Experiments

Development of sub-meV resolution monochromators

High energy-resolution monochromators with $\Delta E/E$ less than 10^{-7} at x-ray energies between 6-30 keV have been under development in our CAT since 1992 [1]. Along these lines, a nested monochromator combining Si (422)-(1064) was developed to operate at 14.4 keV (^{57}Fe Mössbauer resonance energy) with an energy resolution of 5.5 meV. Later, using the same principle, a 15 meV monochromator was developed for ^{119}Sn Mössbauer resonance at 23.87 keV [2]. The ease of tunabality over a wide energy range (i.e., over 100 eV) made these monochromators suitable for both nuclear resonant scattering and inelastic x-ray scattering programs at the SRI-CAT. Members of the X-ray Optics group working at sector 3-ID recently developed a new version in which, instead of using 4-bounce nested geometry, 2-bounce flat asymmetrically cut crystals were employed. With a combination of two Si (975) crystals, 0.9-meV resolution has been obtained at 14.4 keV. This corresponds to $\Delta E/E = 6.2 \times 10^{-8}$. The flux at this bandpass was 4×10^{8} ph/s. We have used this monochromator to measure the phonon density of states of iron metal at room temperature. The experimental setup, raw data as obtained via the inelastic nuclear resonant scattering technique [3], and the phonon density of states are shown in Figure 1.

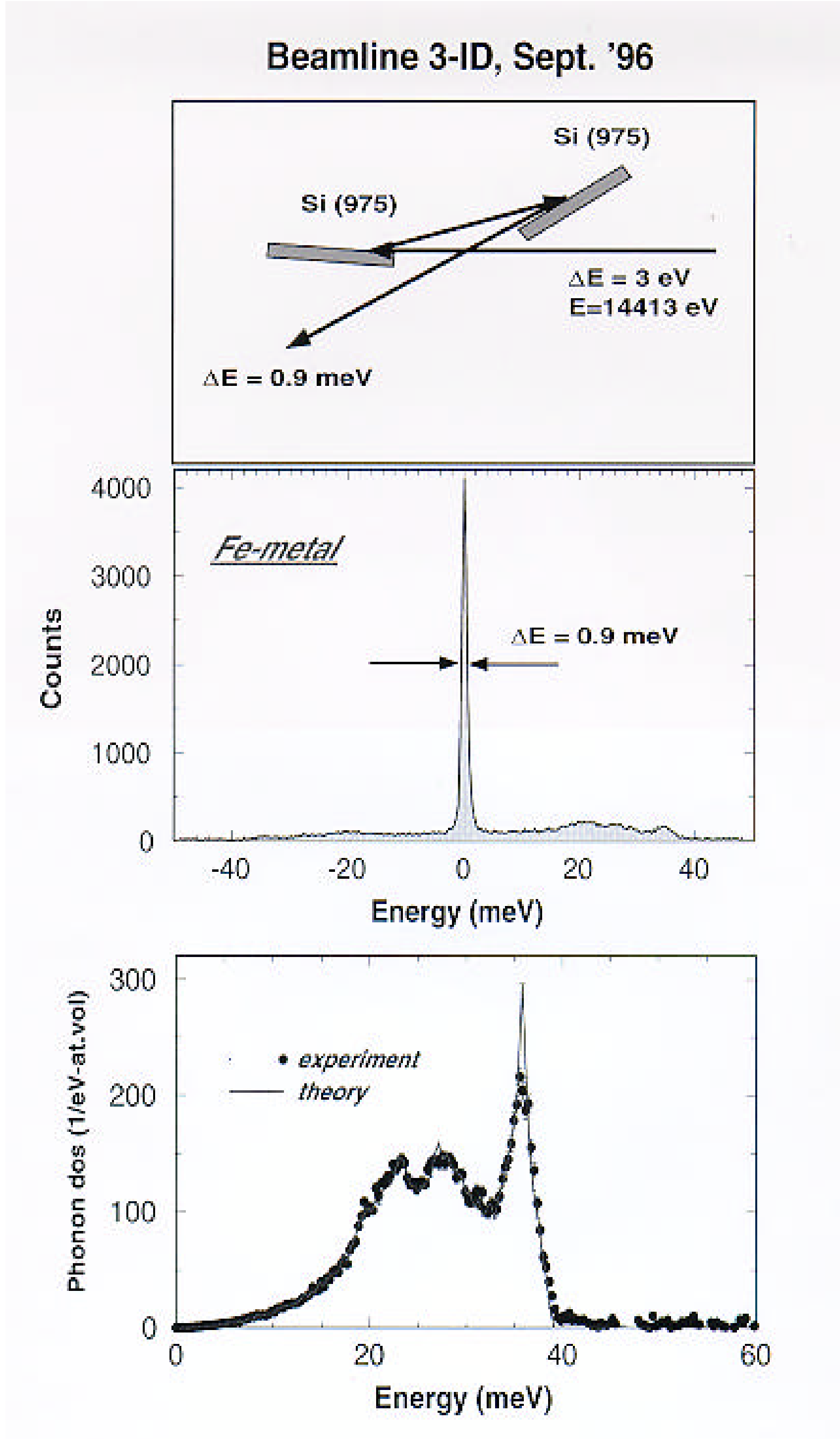

Fig. 1 A schematic of the 0.9 meV monochromator is shown at top. The raw data, as obtained from inelastic nuclear resonant scattering measurement are shown in the middle figure. The energy axis is derived from the angular position of the two crystals. The bottom figure shows the phonon density of states derived from the the raw data. The solid line is a theory curve obtained by using a Born-von Karman analysis to force constants obtained from coherent neutron scattering measurements (courtesy of Prof. B. Fultz, California Institute of Technology).

Measurement of Phonon Density of States in Thin Films at the Advanced Photon Source

Advances in electronic data storage, computers, and telecommunications devices depend on a better understanding of atomic-level two-dimensional structures of materials. These structures include thin films, materials surfaces, interfaces, and artificially layered structures.

Case in point: The study of thin films is essential to the development of new magnetic data-storage media. It is known that the bulk properties of materials are modified when they are prepared in thin-film form. Changes must be measured in order to synthesize films with the desired properties. These studies seek to characterize the atomic structure of thin-film materials by measuring the vibrational frequencies that are determined by the mass of the material's atoms (the atomic weight) and the stiffness of the springs (the bond-force constants) that connect the atoms that make up the material.

Because of the small amount of material in each sample under examination (micrograms or less), techniques like inelastic neutron or x-ray scattering have been difficult to apply. The other available techniques, infrared and Raman spectroscopy, probe only the center of the Brillouin zone due to the low energy and momentum of the incident photons, so they only probe a small fraction of the vibrational states.

High brightness synchrotron radiation sources, such as the Advanced Photon Source, generate tunable, monochromatic x-ray beams that resolve to a few meV. Using the grazing incidence geometry, these beams permit measurement of phonon density of states by inelastic nuclear resonant scattering. This is possible if the angle of incidence is close to the critical angle of the film. The yield may be further improved by choosing a backing with high electron density and can reach close to that of bulk materials (see Figure 2).

The members of the APS X-ray Optics Group, using the 3-ID beamline at the APS, have measured the Fe partial phonon density of states of two amorphous films of Fe_xTb_{1-x}, with x=0.5 and 0.82, each with thickness of 17.5 nm (courtesy of Prof. W. Keune of the University of Duisburg, Germany). The results suggest a softening of thermal vibration (or phonon) modes for the film with the smaller iron content.

The possibility of measuring the phonon modes in thin films brings the opportunity of studying phonon confinement in reduced dimensional systems and leads to a better understanding of thermal and elastic properties of thin films. *E. E. Alp, W. Sturhahn, T. Toellner, R. Roehlsberger, K. Quast, M. Hu, J. Sutter and P. Hession, APS Experimental Facilities Division*

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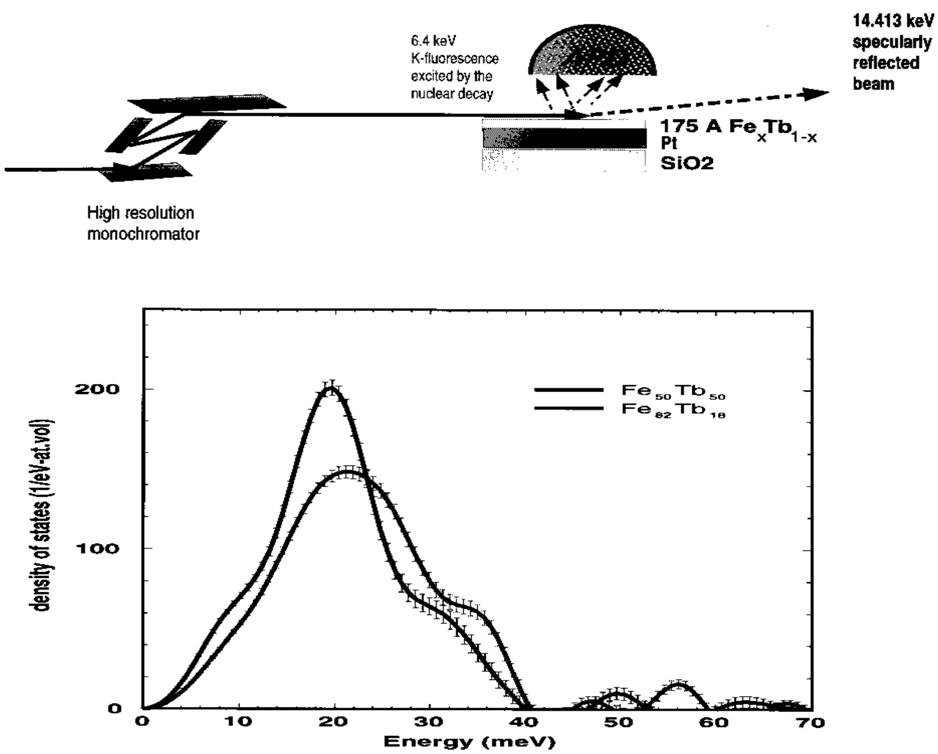


Fig. 2 A schematic of the thin film measurments. The incident beam is monochromatized to 5.5 meV level going through the nested monochromator. The grazing incidence angle is adjusted to increase the yield of the 6.4 keV fluorescence signal. The bottom figure is the Fe partial phonon density of states derived from the data.

The APS Metrology Laboratory and Recent Results

Introduction

A survey of the APS users conducted at the beginning of the year indicated that close to 50 mirrors were to be used in APS beamlines. These will be used for harmonic rejection, focusing, power filtering, or beamline branching. Presently, approximately one third of the mirrors have been ordered, and about a quarter of these 50 mirrors has been received.

State-of-the-art mirror polishing can render surfaces with a sub-angstrom roughness and a sub-microradian rms slope error on small flat substrates of several centimeters in size. As the substrate size increases, or its shape differs from flat (particularly for higher-order surface profiles), the achievable optical quality diminishes. Substrate material and the vendors' polishing technique and expertise are also important parameters in determination of a mirror's quality.

Before being shipped, mirrors are often evaluated by the vendor as part of their quality-assurance process. The Metrology Laboratory at the APS allows for an independent measurement of figure and finish (i.e., slope error and microroughness) as part of the end user's acceptance criteria. The APS Metrology Laboratory (APS-ML) consists of three noncontact optical instruments located in a cleanroom with a tightly controlled environment. Surface microroughnesses on the order of 1 Å rms and slope errors of 1 μrad can be measured with this facility (a detailed description was given in reference 1).

In this article, a basic description of the instruments is presented with the surface qualities of mirrors measured to date. Finally, some future developments are outlined.

The Metrology Laboratory

Instruments

The laboratory has three different instruments to characterize optical surfaces over a range of spatial periods from a few microns up to 2 meters. These instruments are:

- *Long Trace Profiler (LTP)*. The LTP is an instrument designed to measure slope error and curvature of optical surfaces up to 2 m long. It has a sensitivity of 0.1 μrad in slope and 5Å in height, and a reproducibility of better than 0.5 μrad rms. Because of its speed, it offers a rapid and accurate means of evaluating the performance of a mirror bending mechanism. The APS LTP allows measurements of a surface profile of a mirror in either the horizontal or vertical direction. Measurement in the horizontal direction is often desirable in order to eliminate gravitationally induced figure distortion.
- *TOPO Surface Profiler*. The TOPO is a microscope-based instrument that uses visible-light (6303Å) interferometry to measure surface roughness on the order of 1Å. The TOPO profiler can use either a 3D or 2D detector and is currently equipped with three objectives, 1.5 x, 5x, and 40x. Here, 2D refers to the surface height of a line profile. The 3D detector is used when a profile of a surface area (of a few mm^2 depending on the objective) is needed with a minimum resolvable height of 3 Å. The 2D detector obtains a line scan with a resolution of 1 Å along a line up to a few millimeters long, depending on the objective (e.g., 2.05 mm for a 5x objective). The output data can include contour maps, 3D maps, 2D data slices, step-height analysis, power spectral-density and autocovariance functions, and amplitude histogram plots. The TOPO system is designed to handle mirrors up to 1.5 m long and 90 Kg in weight.
- *WYKO 6000 Figure Interferometer*. The WYKO-6000 measures the flatness of optical surfaces interferometrically by a technique similar to that of the TOPO. It is used for probing optical surfaces up to 6" in diameter (or large optics at grazing incidence angles), and can generate 3D profiles of optical surfaces, 2D slices, and Seidel aberration coefficients.

All these instruments are currently operational. Various modifications of the basic systems are being developed for ease of operation and to improve handling of large optics.

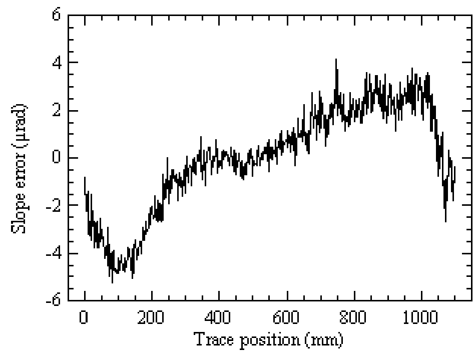
We plan to install a fourth instrument that has both a scanning probe microscope (SPM) and a near-field scanning optical microscope (NSOM) capability. The SPM has both scanning tunneling microscope (STM) and atomic force microscope sensors. These microscopes can be used to obtain topographic maps of surfaces with vertical and lateral resolutions of a fraction of an angstrom. For metrology purposes, these microscopes are very useful for surface roughness measurements. Although the scanned profile lengths are much smaller than needed in order to correlate the SPM measurements with measurements of conventional noncontact profilers, the SPM can be used to understand surface topography better on the atomic scale and relate it to light scattering. In addition, the results can be used to improve methods for making optical surfaces.

Environment

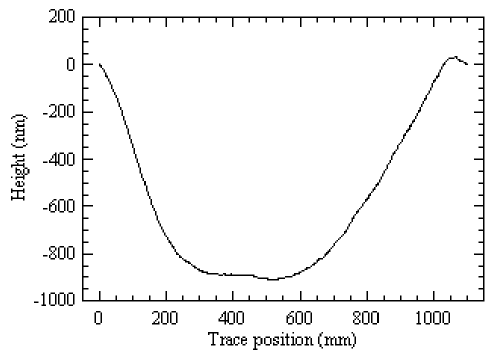
The APS-ML is located in a class-10,000 cleanroom in the APS experiment hall. Mirror handling requires special cleanroom garment and shoe covers, and access to cleanrooms is restricted when measurements are performed.

To ensure repeatability and accuracy of the instruments, the room temperature stability is controlled to better than

(a)



(b)



(c)

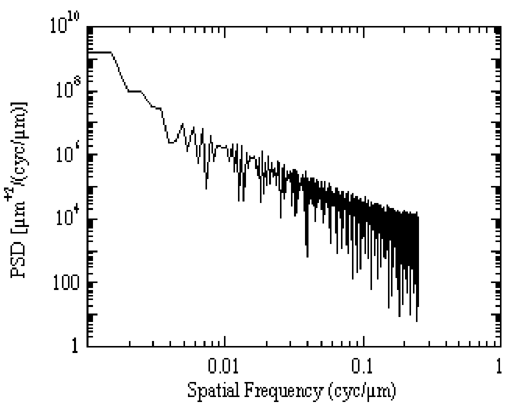


Fig. 1. Example LTP output. Shown are results for the high-heat-load M1 mirror for beamline 2-ID: (a) slope error profile obtained by averaging 5 scans along the mirror center line, (b) corresponding height profile, obtained by integrating the slope error profile, (c) power spectral density of the derived average height profile. The mirror is 1.2 m long and has a slope error of 2.2 µrad rms. The corresponding rms height is 0.21 µm, over a 1100 mm aperture.

+/- 0.5 °C and is regularly monitored. All instruments are mounted on air tables to minimize the effect of vibrations and have the capability of handling large and heavy optics up to 2 m in length and 90 Kg in weight.

Surface quality of mirrors measured to date

In the past two years, seven mirrors, some up to 1.2 m long, have been evaluated in the APS-ML. Two more mirrors (both from ZEISS Corp.) have been delivered recently: a 1.5 m collimating mirror for beamline 1-BM [2], and a 0.8-m-long cylindrical (bendable) mirror for beamline 3-ID [3]. Surface microroughnesses ranging from 1 to 4 Å and slope errors ranging from 1 to 4 µrad have been characterized and found to be consistent with the vendors' measurements. To avoid bias, the vendor's data are obtained after all metrology measurements are completed and analyzed. The results are presented in Table 1 below. These mirrors have been designed and procured by members of SRI-CAT [4-9] and SBC-CAT [10] To date, all of the mirror substrates have been made of either silicon or Zerodur. Most surfaces have at least one reflective coating; platinum and rhodium are typical reflecting materials. For slope error measurement, several lines are typically scanned across the surface of the mirror. In order to minimize instrumental noise, each line is scanned at least five times, then the scans are averaged. Figures 1a, 1b and 1c show a typical output from the LTP. The results are for the 2-ID high-heat-load mirror (see Table 1). Figure 1a shows the slope error profile obtained by averaging five scans on the mirror center line. Figure 1b gives the corresponding height profile obtained by integrating the slope error profile. Figure 1c shows the power spectral density of the derived average height profile. The power spectral density is useful for computing the rms statistics over a selected spatial frequency bandwidth. This allows one, for example, to separate "mid-frequency ripple" from over all surface figure. Further discussion on the usefulness of the power spectral density calculation is given below.

To check the consistency of the measurements, the mirror is usually rotated 180°, and the process is repeated. It is sometime desirable to have a 3D profile of an optical surface. This can be done by scanning a large number of lines on the surface and then connecting the resulting profiles in the transverse direction to generate a 3D profile of the mirror's surface. This is useful, for example, to predict the performance of a mirror using a ray-tracing code such as SHADOW [11].

Optical surface roughness is typically measured on several sites chosen randomly to avoid periodicity due to the manufacture's polishing technique. Before measurements, the TOPO is checked for calibration and repeatability. As with the LTP, each site is measured several times, and the results are averaged to minimize error due to the system's noise.

Power spectral density

It is well known that scattering is the main effect of the imperfect surface finish of optical surfaces. The amplitude of the scattering depends on the power spectral density (PSD) of the surface roughness, which gives heights or slope error distribution as a function of their spatial frequencies. For a one-dimensional profile, the PSD can be written [12]:

$$S(f) = \lim_{L \to \infty} \frac{2}{L} \left| \int_{-L/2}^{+L/2} \exp(i2\pi f x) Z(x) dx \right|^2$$

where L is the profile length, f is the spatial frequency, and Z (x) is the height at a position x.

An interesting feature of the PSD is the fact that surface statistics parameters, such as rms roughness, rms slope error and rms curvature, can be derived from it by evaluating its first, second, and fourth moments, over a selected spatial frequency bandwidth. The PSD curve can also be used to predict the absolute intensity and shape of the angle-resolved scattering curve at x-ray wavelengths, and its value can be directly related to the amount of light scattered into a given solid angle over a defined bandwidth.

Future developments

Currently, we are working on the following ways to enhance the capability and performance of the APS-ML:

- Refinement of mirror measurement procedures and preparation of standardized results documents.
- Development and maintenance of the mirror measurements database. For future reference and design considerations, a mirror measurement database is being developed. The database will contain a variety of information, including metrology results, design parameters and specifications.

Sector - Beamline	Vendor	Length (m)	Shape	Specifications			Vendors' Metrology			APS Metrology		
				Δα (μrad)	Δz (Å)	R (m)	Δα (μrad)	Δz (Å)	R (m)	Δα (μrad)	Δz (Å)	R (m)
19 - BM	Rockwell	1.02	Flat bendable	1.0	2.0	600.0-∞	0.9	2.0	-	1.0	2.3	Measured flat
2 - ID	Rockwell	1.20	Flat	<4.0	4.0	∞	2.2 /	2.1	-	2.2	2.7	-
2 - BM	Rockwell	0.88	Flat	<3.0	<4.0	∞	2.0	2.4	-	2.1	2.4	-
2 - ID C	Continental Optical	0.24	Spherical	1.0	5.0	448.7±1 %	Not Avail.	Not Avail.	-	0.8	<2.0	448.2
2-ID-C	Continental Optical	0.18	Spherical	1.0	5.0	171.9±1 %	Not Avail.	Not Avail.	-	1.1	2.0	172.4
2 - ID B	General Optics	0.25	Spherical	<3.0	≤2.0	800.0±3 %	Not Avail.	0.58	793.3	0.9	1.1	820.4
1 - BM	SESO	0.55	Flat	<1.0	≤5.0	∞	2.9	2.5	-	2.9	2.8	-

Table 1. Metrology results of mirrors delivered and characterized to date. Δa is the rms slope error, Δz is the rms roughness, and R is the radius of curvature.

- Further improvement of the metrology environment (air flow, vibration, and temperature stability) in order to achieve better instrument repeatability.
- Installation of the NSOM-SPM system.
- Other plans under consideration include: (1) develop *in-situ* measurement techniques, (2) develop x-ray characterization techniques for mirrors, in order to establish correlation with measurements obtained by conventional visible light instruments, (3) establish, in a collaboration with other facilities, a calibration standard for the LTP, (4) investigate polishing requirements and techniques to preserve the coherence of the high brilliance x-ray beam reflected from x-ray mirrors, (5) establish a close interaction between the APS-ML and the optical fabrication facility in order to improve mirror polishing technique.

Acknowledgments

We wish to thank our colleagues at the APS, the users, and staff at other facilities for their assistance. We particularly thank A. M. Khounsary for his valuable help with some of the measurements, Peter Takacs (of BNL), Cindy Bresloff (currently at Tinsly Laboratories, Inc.), and Jean Susini (of ESRF) for numerous and helpful discussions, and R. Hopf, D. Fergusson, W. McHargue, and J. Arko for technical assistance. This work is supported by the U.S. Department of Energy, BES - Material Sciences, under contract number W-31-109 ENG-38. *L. Assoufid and D. M. Mills, APS Experimental Facilities Division*

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Calendar of Events

March 17-21,1997
1997 APS March meeting
Kansas City, MI

May 12-16 ,1997
1997 Particle Accelerator Conference
Hotel Vancouver, Vancouver, B.C., CANADA

June 17-20 ,1997
1997 National Conference on Synchrotron Radiation Instrumentation
Cornell University, Ithaca, NY

July 27 - August 1, 1997
Sagamore XII - Charge, Spin and Momentum Densities
Prince Albert National Park, Saskatchewan, CANADA

July 27 - August 1, 1997
SPIE International Symposium on Optical Science, Engineering, and Instrumentation
San Diego Convention Center, San Diego, CA

August 3-8 ,1997
Gordon Research Conference on X-ray Physics
Plymouth State College, Plymouth, NH

August 4-8,1997
6th International Conference on Synchrotron Radiation Instrumentation (SRI 97)
Himeji Citizens Hall, Hyogo, JAPAN

Publications

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